INNOVATIVE FABRICATION TECHNIQUES FOR AEROSPACE PROPELLANT AND PRESSURANT TANKS

SBIR PHASE I

CONTRACT NAS3-98104

FINAL REPORT

ELECTROFORMING RESEARCH AND DEVELOPMENT GROUP

ELECTROFORMED NICKEL, INC.

785 MARTIN ROAD

HUNTSVILLE, ALABAMA 35824

BY:

G. A. MALONE RICH EDWARDS BRIAN BABCOCK WANDA HUDSON

14 JULY 1999

PREPARED FOR:

NASA - JOHN GLENN RESEARCH CENTER TECHNOLOGY SUPPORT BRANCH 21000 BROOKPARK ROAD CLEVELAND, OHIO 44135

ACKNOWLEDGEMENT:

THIS EFFORT IS FUNDED BY THE BALLISTIC MISSILE DEFENSE ORGANIZATION SMALL BUSINESS INNOVATION RESEARCH PROGRAM AND ADMINISTERED BY THE NASA - JOHN GLENN RESEARCH CENTER

DTIC QUALITY INSPECTED 4

DISTRIBUTION STATEMENT A
Approved for Public Release
Distribution Unlimited

19991110 039

PROJECT SUMMARY

To be economically attractive, weight and performance for small earth-to-orbit launch systems and station keeping space vehicles must be improved at significantly lower costs while maintaining the required payloads. A major weight and cost factor in any of these craft is the tankage for propellants and pressurant gases. Innovative and proven manufacturing technologies such as electroforming have often been overlooked as means to meet these needs. Electroforming offers a method to fabricate seamless tanks with no property degrading welds. In conjunction with filament winding, electroforming can produce improved tanks in greatly reduced time frames because it can produce the material and net shape simultaneously. For those readers not familiar with electroforming, it is a plating process whereby a mandrel (or form) is made conductive and overplated with a metal such as nickel or copper. The mandrel is later removed chemically or thermally. It is widely used in fabricating rocket thrust chambers such as the Space Shuttle MCC (Main Combustion Chambers) and the Shuttle OME (Orbital Maneuvering Engines).

Although electroforming nickel based alloys of very high strength was initially planned, discusions with tankage engineers from prominent aerospace companies indicated that material with high yield and ultimate strength might not be desired. Instead, a highly ductile electrodeposit was sought which could easily expand under pressure to intimately contact an overlying filament wound shell made from a very high modulus material. The shell thus provides the structural pressure containment capability while the metal tank liner provides a seal to prevent leakage while integrating the fill and drain ports into a singular structure. Since nickel-cobalt electrodeposits have low ductility as deposited and require stress relieving treatments at temperatures higher than desired for the binding system in the filament winding, a need to direct development efforts to high ductility nickel was required. This task was extremely important in that high ductility would be required in very thin deposits since these would be required if weight savings were to be realized.

Task I addressed the ductility problem. It was found that best ductility (10 percent elongation, or more, in 2 inch gauge lengths) could be obtained in deposits having very fine (almost equi-axed) grains. Ductilities were also determined in 1 inch gauge lengths since nickel electrodeposits tend to elongate locally in many cases. The results of these tests were even more encouraging. Stress relieving at 177°C (the filament wind "cure" temperature) generally improved the ductility. Electrodeposition parameters for producing this fine grain were mapped. Discussions with tank design engineers indicated that these properties would be more than adequate.

Since electroforming of large tanks and tank liners might be facilitated by electroform joining ("cold welding") of tank components, Task II efforts demonstrated that excellent joint strengths could be obtained. Such strength results (obtained in mechanical pull tests) were similar to the mechanical strength of the two electroformed metals being joined. This was a far superior strength to that obtained by thermal means. The properties of the electroformed metals were not degraded. Application of this joining technique was demonstrated on two halves of an electroformed nickel tank having a wall thickness of about 0.038 cm (0.015 inch). The "cold welded" tank was leak tested at

about 200 psig and performed very well. This demonstrator tank has caught the interest of local companies and NASA - MSFC engineers as a highly innovative means for producing economical light-weight tankage.

As an alternate to nickel as a tank liner material, we addressed the possibility for electroforming titanium from a non-aqueous electrolyte. One advantage for titanium might be in the containment of storable propellants such as nitrogen tetroxide which (if wet) will attack nickel. Although a functional cell was set up in an argon atmosphere, the deposits were rather powdery and non-coherent. We believe this system can be made to work, but better stability of the bath must be obtained.

Since many tank designers desire to filament wind the outer high modulus jacket first, we examined the task of electroforming nickel onto cured carbon filament samples. These samples were tested by cycling though cryogenic fluids (much as they would be required to do in flight tankage). Adhesion of the nickel to the carbon composite materials was very good.

1.0 PROJECT OBJECTIVES

Ę

The Phase I technical objectives to be met in demonstrating the merits of this innovation to produce high performance pressurant and propellant tanks more economically than conventional forming and welding techniques were as follows:

1.1 Task I - Demonstration of Mechanical Properties of Electroformed Nickel of Various Thicknesses

Initially this task was intended to demonstrate high tank wall strengths obtainable with electroformed nickel-cobalt alloys and compare these results with conventional unalloyed nickel deposits. However, consultations with tank designers indicated that ductility was more desireable than high strength, since the high pressure loading of the containment vessel would be carried by the outer shell of filament wound material having a much higher modulus. This redirected our efforts to evaluation of conventional nickel electrodeposits over a wide range of thicknesses and deposition parameters and determining the effects of post winding cure temperatures on these properties.

Electroforming bath temperatures, bath pH, and deposition rates (current densities) were to be evaluated for various tank liner thicknesses anticipated for flight hardware. Mechanical property test results for "as-deposited" and 177°C heat treated samples were to be obtained. The 177°C cure temperature for the filament winding was based on advice from tank design and manufacturing engineers. Metallographic examinations were to be made to determine if microstructural similarities existed between specimens showing best ductility.

1.2 Task II - Demonstration of Electroform Joint Integrity

Simple flat electroformed nickel sheet samples are to be joined by electroforming (a process known as "cold welding" demonstrate the unusual bond strengths obtainable by this process. Strengths approaching those of the materials being joined are sought. The process shall be demonstrated to be applicable to tank hardware by using it to join two electroformed tank halves and testing the vessel to assure joint integrity (absence of leaks).

1.3 Task III - Demonstration of Non-aqueous Titanium Electrodeposition Process

A non-aqueous titanium electrodeposition electrolyte is to be established in an argon atmosphere and plating trials conducted to establish feasibility of the process. Bath temperature, titanium concentration, and current density effects are to be studied.

1.4 Task IV - Investigation of Coherent Electrodeposits on Carbon Filament Tank Shells

Specimens of cured carbon fiber layups are to be surface treated to improve surface adhesion characteristics and nickel plated to demonstrate an alternate approach to making composite tanks with electroformed sealing liners. Evaluation shall be by a method that shall verify a good adhesion

2.0 PROJECT WORK CONDUCTED AND RESULTS OBTAINED

2.1 Task I - Demonstration of Mechanical Properties of Electroformed Nickel of Various Thicknesses

Three 136 liter electroforming tanks were used for this task. Each tank contained a conventional sulfamate nickel electrolyte originating from one common solution to assure that no differences in chemical composition would exist. The tanks were designated Tank A, Tank B, and Tank C with the pH of Tank A set and maintained at 3.6, Tank B at 4.0, and Tank C at 4.4. Nickel metal in each tank was 82.4 g/l, boric acid was 31.5 g/l, and chloride concentration (for proper anode corrosion) was 0.40 g/l. Samples were electroformed from each tank at temperatures of 46.1°C (115°F), 48.9°C (120°F), and 51.7°C (125°F). Using conventional dc power supplies, samples for each bath pH and bath temperature were produced at current densities of 20, 30, and 40 amperes per square foot. For some samples, pulsed or pulsed-periodic current reversal was used to determine effect on grain structure. Periodic solution analyses were made and pH adjustments made as necessary to control to +/- 0.2 pH units for each tank. Each tank was constructed with a weir to provide a compartment for continuous filtration and to make additions when necessary. Arrangements were made for automated water additions to the weir compartment 24 hours per day to maintain solution volume and chemistry ranges. Figure 1 illustrates a typical tank.

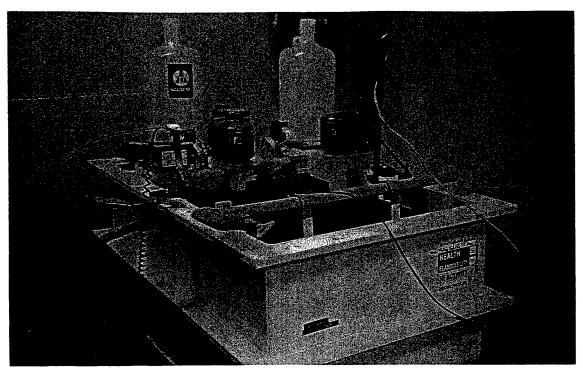


Figure 1. Typical Sulfamate Nickel Electroforming Tank and Support Equipment.

The plating compartment of each tank contained a quartz heater with a temperature control capable of maintaining +/- two degrees Fahrenheit. Each plating compartment was also equipped with a pump designated to supply a forced flow of electrolyte perpendicular to the surface of the mandrel on which nickel specimens were deposited. Stainless steel sheet mandrels were used so that thin samples could be easily removed. Each mandrel was contained in a plastic box shield designed to promote uniform deposit thicknesses over the entire sample. This was necessary to obtain nickel deposits that would react uniformly during mechanical property testing. To assure that proper current was applied to electroform each sample, Yokogawa proimary standard quality ammeters were used in series with the power supplies. Current settings were made based on readings from these instruments.

All specimens were cut into strips suitable for machining test bars or foils in accordance with ASTM E-8. All mechanical property testing was subcontracted on purchase orders to the same testing house. Very thin specimens were electric discharge machined into the required test strips. This assured freedom from burrs or cold working which might affect test results. The same firm that performed the mechanical property testing did the metallographic work. Photomicrographs at 100X magnification were supplied for each sample to illustrate grain size and configuration.

Appendix A references all test information for nickel electrodeposits having thicknesses greater than 0.071 cm (0.028 inch). For the pH 3.6 bath, best ductility using conventional dc power appeared to occur at the higher bath temperature of 51.7°C and the lower current density of 20 ASF. For pH 4.0 baths there appeared to be no specific parameters promoting better ductility; most values at lower current densities appeared acceptable. At a pH of 4.4 and conventional dc power, the better ductilities seemed to occur at the lower bath temperature. Use of pulsed current appeared to be beneficial for ductility in general; however, pulse with periodic current reversal provided many of the best ductility results over all temperatures and pH values. Test results for stress relieved companion samples are shown for comparison. Stress relief was at 350°C (650°F) for one hour. This was much higher than the 177°C expected for tank liners. However, the thicknesses of these samples were greater than would normally be used on tank liners. Stress relieving almost always resulted in improved ductility.

Appendix B summarizes similar test data for nickel electrodeposits produced at thicknesses of 0.0254 to 0.0508 cm (0.010 to 0.020 inch). For this thinner material, the ductilities were reduced as expected. The stress relief at 177°C did not result in much improvement. Acceptable ductilities were found generally at low current densities of 20 ASF and higher bath temperatures. Pulsed current was not used on this group of samples.

Limited data, Appendix C, for very thin nickel deposits (0.004 to 0.006 inch thickness) indicated that high bath temperature and low current density or moderate bath temperature with pulsed current with periodic current reversal would provide acceptable ductility. From this data it is reasonable to predict similar benefits to deposits in the 0.010 to 0.020 inch thickness range. Retainer samples of most of these deposits are available for additional studies if required.

Conclusions

- Conventional unalloyed nickel deposits can be produced in any thickness with the ductility deemed suitable for pressurant or propellant tank liners to be overwrapped with composite fibers of high modulus.
- Low current densities and higher bath temperatures generally improve ductility.
- Pulsed curret, and especially pulsed current with periodic reverse, appears to improve ductility significantly.
- Good ductility is more difficult to produce in very thin electroformed nickel than in thicker material. This was anticipated and may be due to the initial effects of substrate on the microstructure of deposits.

2.2 Task II - Demonstration of Electroform Joint Integrity

Flat electroformed nickel specimens were produced at thicknesses of approximately 0.005, 0.010, and 0.015 inch thicknesses. Each sheet was cut into two pieces and each piece was butted against its companion piece of similar thickness for electroform joining. A small gap was left between each piece. The gap was made conductive with silver pigmented paint, and excess paint was sanded from the individual flat components. A small shield was placed around each specimen to control deposit thickness variations. The adjacent nickel surfaces were anodically treated in 25% by volume sulfuric acid at room temperature to remove any surface cold work. The samples were then rinsed well. The next operation was to cathodically activate the nickel for subsequent electroform bonding. This was performed in 25% sulfuric acid at a current density of 100 ASF for at least 3 minutes. The samples were rinsed in sulfamic acid for compatibility with the plating bath and to maintain acidity of the surface to deter oxidation. They entered the electroforming bath with voltage applied so as to start plating immediately. At least 0.010 inch thickness of nickel was applied to the "cold weld" joint. Strips were then cut out so as to produce tensile test bars with the electroform weld as a part of the cross-section. Testing was performed with the following results:

COLD WELD TENSILE TEST RESULTS

Sample No.	Thickness (in)	Yield Str. (Ksi)	Tensile Str (ksi)	Elong. In 2 in, %
CF 4005	0.0047	116	140	6
CF 4010	0.0093	104	128	7
CF 4015	0.0130	121	149	3.5

A thin wall (approximately 0.012 inch thickness) containment vessel was electroformed over a mandrel machined from high density polystyrene. A hole was machined through the center of the mandrel in a direction parallel to the major axis of the tank with location at the center of this major axis. This provided means for attaching stainless steel fill/drain ports at each end with the ability to

hold these ports firmly in place with lock-nuts. The fittings (ports) were masked so that the mandrel could be spray painted with a silver pigmented conductive coating. The masking was removed from the ports and the entire assembly was mounted on a horizontal rotating fixture equipped with a spring loaded contact rod to pass current to the metal ports and hence to the silver conductive coating. The rotating fixture contained a variable speed drive motor electrically isolated from the part by plastic drive gears.

Once the tank liner was electroformed, the tank liner was carefully cut in half at the center of the cylindrical section. The liner halves were physically removed from the mandrel. A new cylindrical plastic mandrel section was made onto which the two tank halves were abutted. Silver paint was applied to seal the openings in the "cold weld" gap, and excess silver removes by sanding with fine grit paper. The tank halves not to receive electrodeposit were tape masked. The exposed surfaces were cleaned and activated by the previously described means. After suitable electroforming time for a closeout, the part was inspected and reworked as needed until visually acceptable. The mandrel was dissolved and the part subjected to hydrostatic pressurization to about 200 psig. The reworked part showed no leakage and was considered a success. Figure 2 illustrates various stages of the electroform "cold welding" utilized in this task.

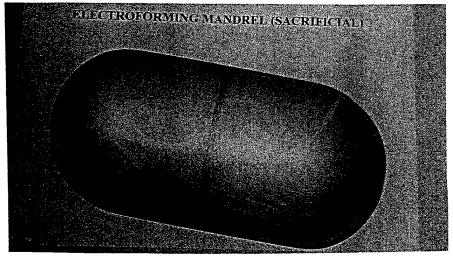
Conclusions

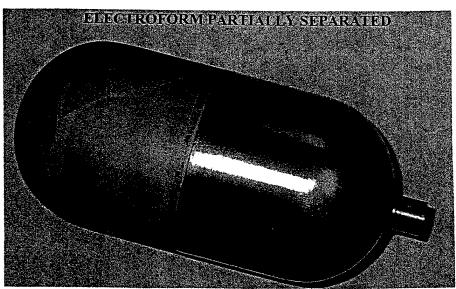
- Electroforming can be used as a very effective means for joining two metals together without the use of thermal means. This preserves material properties since there is no heat affected zone.
- Electroform "cold welding" has been successfully demonstrated in joining two tank halves together with a high integrity joint.

2.3 Task III - Demonstration of Non-aqueous Titanium Electrodeposition Process

Titanium can not be electroplated from aqueous (water containing) electrolytes because water will electrolytically break down before the deposition potential of titanium is reached. This necessitates the use of non-aqueous electrolytes such as used to electrodeposit aluminum. Deposition must be conducted in an atmosphere containing no oxygen or water. For this task a glove box was used to house the electroplating facility. Argon was supplied to the glove box through feed ports in the glove box wall to expell oxygen. The glove box interior was kept at a slightly positive pressure using the Argon gas. The plating apparatus consisted of a hotplate, a container for a non-aqueous liquid to serve as a heating jacket for the beaker in which plating was to be conducted, and a power supply located outside of the glove box and having electrical leads attached to positive and negative posts mounted through the glove box wall. A dessicant container was attached in series to the Argon supply line to reduce chances of introducing water to the box interior. An illustration of the facility is found in Figure 3.

A one liter volume of electrolyte was prepared by adding reagent grade titanium tetrachloride





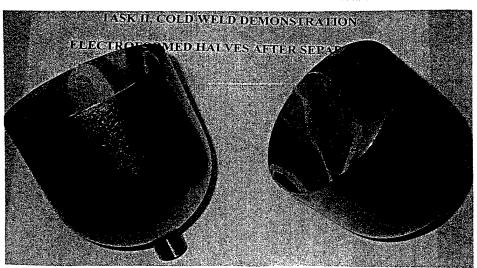
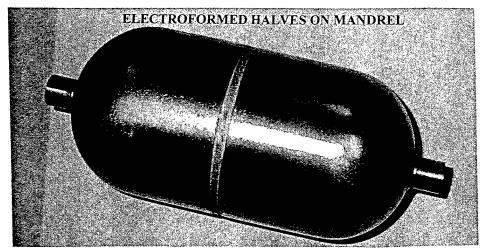
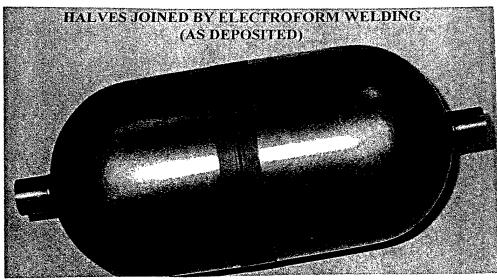


Figure 2a. Processing Sequence for Electroformed "Cold Weld" Joining of Tank Components





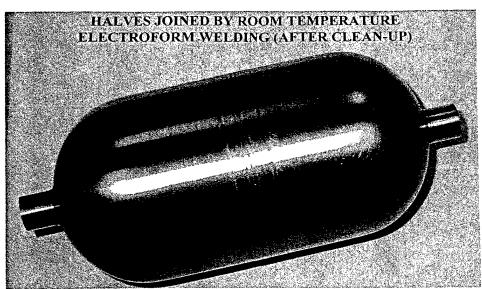


Figure 2b Processing Sequence for Electrformed "Cold Weld" Joining of Tank Components

to a solvent composed of 80 percent by volume toluene and 20 percent by volume ethylbenzene. This electrolyte is similar to the baths used by Caleardi and Capuano in their studies of aluminum plating solutions. All required chemicals and equipment were placed in the glove box which was sealed to prevent entry of airborne moisture and oxygen. The glove box atmosphere was purged with argon for several hours. Titanium tetrachioride (Aldrich, 99.9%) was added to the organic electrolyte to produce a solution of 0.5 molarity TiCl₄. Upon opening the titanium tetrachioride, a small amount of Hcl formation was noted indicating that a trace of air was still in the enclosure. Since it is desired to electrodeposit titanium from a valence state lower than four, a reduction process was needed. A small amount of magnesium powder was added to the electrolyte to reduce most of the titanium to the plus two valence. The solution was expected to turn to a yellowish color; however, a red-orange color was noted along with some precipitation believed to be titanium dioxide. This settled to the bottom of the plating vessel which was a one liter heavy wall flask.

Initial plating attempts did not yield any significant titanium deposits due to the great difficulty in obtaining suitable conductivity in the electrolyte. A conductivizing salt was needed. Lithium chloride was added but the solubility was limited and little improvement in conductivity was noted. Addition of lithium acetate was more successful. More plating trials were made but any deposits obtained were either unstable or of powdery, non-coherent quality. Figure 3 illustrates the glove box facility.

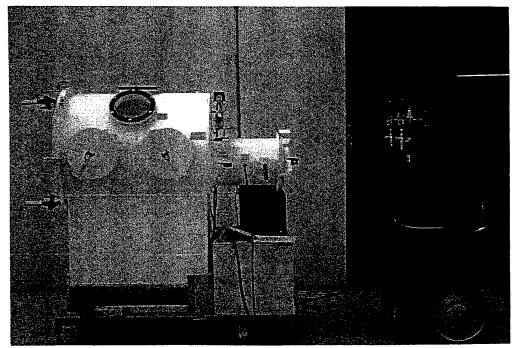


Figure 3. Glove Box Facility in Which Titanium Plating Trials Were Conducted.

Conclusions

An electrolyte for deposition of titanium using toluene-ethylbenzene is feasible. It can be formulated with suitable conductivity.

Better protection of the facility to exclude moisture and oxygen is needed. Use of in-line dessicants and greater argon or nitrogen purge rates are required. It may also be preferred to use titanium chloride premixed with toluene to prepare the bath. This decreases the risk of contamination when mixing the electrolyte. Direct argon or nitrogen purging of the plating vessel (in addition to the glove box) might be beneficial. It would also be preferred to use magnesium ribbon instead of the powder which is prone to surface oxidation.

2.4 Task 4 - Investigatrion of Coherent Electrodeposits on Carbon Filament Tank Shells

Samples of cured carbon filament wound plates were supplied by GenCorp Aerojet for nickel plating to evaluate bond strength. Each specimen was 2 inches by 3 inches and about 0.150 inch in thickness. Surface preparation consisted of sanding the surface to be plated using 320 grit metallurgical paper. Only one surface was prepared and the backside was masked. The sanded surface was detergent cleaned and rinsed to remove any particulate matter from the prepared surface. Nickel was deposited from a conventional sulfamate bath operated at 47.8°C (118°F) starting at 0.54 A/dm² (5 ASF). The current was gradually increased to 2.15 A/dm² (20 ASF) until at least 09.005 inch of nickel was deposited. Three samples were thus electroplated and submitted to Gencorp Aerojet for evaluation.

Testing consisted of thermal shock cycling whereby samples were submerged in liquid nitrogen, withdrawn, and recycled for a number of times. There were no signs of separation of the deposits from the carbon filament substrates.

Conclusions

• Nickel sealant liners can be electrodeposited on interiors of carbon filament wound outer shells to provide an alternate means of fabricating propellant tanks and pressure vessels. The adhesion of such deposits is suitable for use in vessels requiring cryogenic cycling.

3.0 Recommendations for Future Studies and Demonstrations

- Demonstrate that baffles can be integrated into electroformed tank liners to minimize sloshing at a minimal cost.
- Evaluate inexpensive means to produce tank liner mandrels which are presently a major time and cost restraint in electroforming tank liners.
- Show that lighter weight fill and drain ports can be integrated into the tank liner with good structural integrity.
- Continue the titanium deposition effort using improved atmospheric control measures.

APPENDIX A

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR AS-DEPOSITED AND STRESS RELIEVED NICKEL DEPOSITS FROM SULFAMATE BATHS - DEPOSIT THICKNESSES IN EXCESS OF 0.071 CM (0.028 INCHES)

1998 STUDY IN 136 LITER BATHS

Chemistry	Sample A115-20	Sample A115-30	Sample A115-40	
Nickel Metal, g/l	82.4	82.4	82.4	
Bonc Acid. g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
рН	3.6	3.6	3.6	
Bath Temperature, °C	46.1	46.1	46.1	
Filtration, Micron Rating	20	20	20	
Current Mode	Conventional dc	Conventional de	Conventional dc	
Current Density, A/dm ²	2.15	3.23	4.30	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited			0.110	
Thickness, Cm	0.117	0.071	0 112	
Ultimate Strength, ksi	114.80	78.02	93.01	
Yield Strength, ksi	65.83	52.97	62.16	
Elongation in 5.08 Cm. %	7.5	12.5	7.5	
Vickers Hardness, 100 g Load	206	181	206	
Stress Relieved at 350°C for 1 Hou				
Thickness, Cm	0.122	0.074	0.113	
Ultimate Strength, ksi	73.22	71.68	77.75	
Yield Strength, ksi	44.77	54.02	55.28	
Elongation in 5.08 Cm, %	21.5	9.5	10.5	

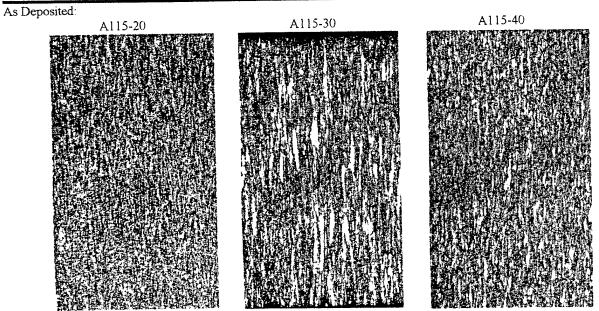


Figure A-1. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Conventional Current.

Chemistry	Sample A115-20A	Sample A115-20B	Sample A115-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
рН	3.6	3.6	3.6	
Bath Temperature, °C	46.1	46.1	46 1	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle		
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average		2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
as-Deposited				
Thickness, Cm	0.107	0.140	0.112	
Ultimate Strength, ksi	104.60	120.70	104.40	
Yield Strength, ksi	61.55	82.55	67.78	
Elongation in 5.08 Cm, %	6.0	8.5	13.5	
Vickers Hardness, 100 g Load	221	221	206	
tress Relieved at 350°C for 1 Hour			200	
Thickness, Cm	0.109	0.137	0.112	
Ultimate Strength, ksi	69.48	90.74	84.59	
Yield Strength, ksi	40.30	59.76	56.70	
Elongation in 5.08 Cm, %	22.50	14.0	20.0	

Photomicrographs (100 X Magnification)

As Deposited:

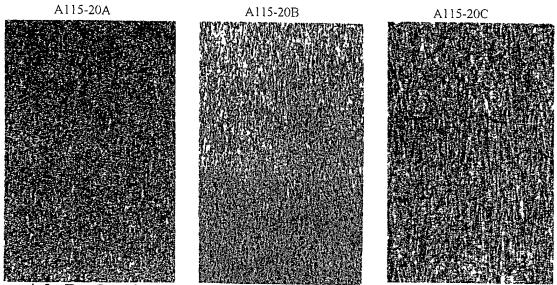


Figure A-2. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample A115-30A	Sample A115-30B	Sample A115-30C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
рН	3.6	3.6	3.6	
Bath Temperature, °C	46.1	46.1	46.1	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 30% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	3 On, 7 Off	8 Forward, 2 Reverse	
Current Density, A/dm²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.102	0.099	0.058	
Ultimate Strength, ksi	72.00	79.82	92.33	
Yield Strength, ksi	46.50	52.78	73.17	
Elongation in 5.08 Cm, %	17.5	12.5	9.5	
Vickers Hardness, 100 g Load	177	181	206	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.099	0.104	0.056	
Ultimate Strength, ksi	70.62	70.94	73.37	
Yield Strength, ksi	52.20	47.38	52.08	
Elongation in 5.08 Cm, %	10.0	20.5	15.0	

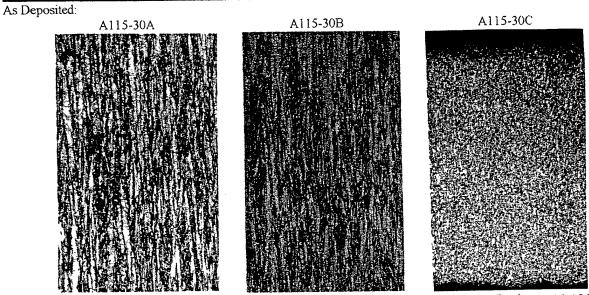


Figure A-3 Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample A120-20	Sample A120-30	Sample A120-40
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	. 31.5
Chloride, g/l	0.406	0.406	0.406
Perating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
s-Deposited			
Thickness, Cm	0.150	0.124	0.114
Ultimate Strength, ksi	108.10	102.00	93.18
Yield Strength, ksi	70.17	73.88	66.84
Elongation in 5.08 Cm, %	15.0	5.0	6.5
Vickers Hardness, 100 g Load	193	221	190
stress Relieved at 350°C for 1 Hou			
Thickness, Cm	0.152	0.130	0.107
Ultimate Strength, ksi	87.82	85.16	88.81
Yield Strength, ksi	65.74	54.30	62.35
Elongation in 5.08 Cm, %	20.5	12.0	6.5

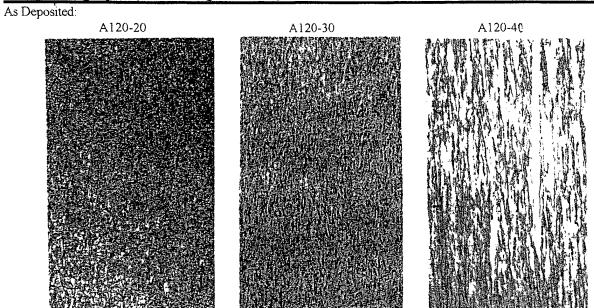


Figure A-4. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

Chemistry	Sample A120-20A	Sample A120-20B	Sample A120-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Bone Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pН	3.6	3.6	3.6	
Bath Temperature, °C	48.9	48.9	48.9	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.152	0.152	0.114	
Ultimate Strength, ksi	98.08	97.03	99.21	
Yield Strength, ksi	62.62	65.68	62.17	
Elongation in 5.08 Cm, %	10.0	9.0	12.0	
Vickers Hardness, 100 g Load	228	206	213	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.147	0.140	0.107	
Ultimate Strength, ksi	79.54	74.69	86.03	
Yield Strength, ksi	56.45	54.72	58.94	
Elongation in 5.08 Cm, %	15.0	22.0	19.0	

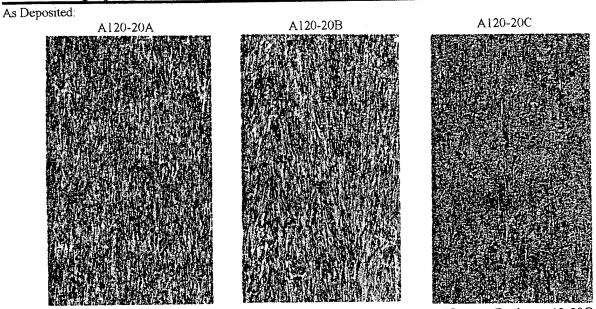


Figure A-5. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample A120-30A	Sample A120-30B	Sample A120-30C
Nickel Metal, g/l	82.4	82.4	82.4
Borne Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On. 5 Off	2 On. 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.135		
Ultimate Strength, ksi	75.02		
Yield Strength, ksi	46.89		
Elongation in 5.08 Cm, %	16.5		
Vickers Hardness, 100 g Load	187		
Stress Relieved at 350°C for 1 Hou	r		
Thickness, Cm	0.132		
Ultimate Strength, ksi	71.35		
Yield Strength, ksi	49.60		
Elongation in 5.08 Cm, %	12.5		

Photomicrographs (100 X Magnification)

As Deposited.

A120-30A A120-30B A120-30C

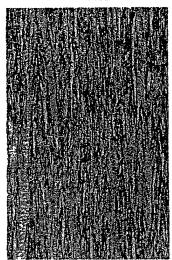


Figure A-6. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample	A125-20	Sample A125-30	Sample A125-40
Nickel Metal, g/l		32.4	82.4	82.4
Boric Acid, g/l		31.5	31.5	31.5
Chloride, g/l		406	0.406	0.406
Operating Conditions				
рН		3.6	3.6	3.6
Bath Temperature, °C		51.7	51.7	51.7
Filtration, Micron Rating		20	20	20
Current Mode	Conve	ntional dc	Conventional dc	Conventional dc
Current Density, A/dm ²		2.15	3.23	4.30
Anodes) Ni	SD Ni	SD Ni
Solution Agitation		oray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties				
As-Deposited	(1)	(2)		
Thickness, Cm	0.147	0.145	0.119	0.130
Ultimate Strength, ksi	107 20	105.90	97.92	75.15
Yield Strength, ksi	75.86	68.51	63.59	48.14
Elongation in 5.08 Cm, %	15.5	14.5	10.0	12.5
Vickers Hardness, 100 g Load	228	216	225	147
Stress Relieved at 350°C for 1 Hou				
Thickness, Cm	0.145	0.142	0.122	0.119
Ultimate Strength, ksi	89.48	86.24	81.61	85.30
Yield Strength, ksi	57 67	59.29	53.85	59.82
Elongation in 5.08 Cm, %	16.5	18.0	12.0	7.5

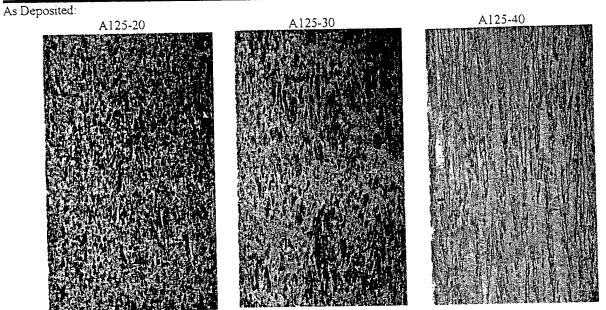


Figure A-7. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Conventional Current.

Chemistry	Sample A125-20A	Sample A125-20B	Sample A125-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pH	3.6	3.6	3.6	
Bath Temperature, °C	51.7	51.7	51.7	
Filtration, Micron Rating	20	20	2 0	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On. 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.145	0.117	0.114	
Ultimate Strength, ksi	102.70	108.50	101.60	
Yield Strength, ksi	70.66	70.44	67.14	
Elongation in 5.08 Cm, %	9.0	10.0	13.0	
Vickers Hardness, 100 g Load	222	221	206	
Stress Relieved at 350°C for 1 Hou				
Thickness, Cm	0.150	0.117	0.112	
Ultimate Strength, ksi	83.89	84.05	81.15	
Yield Strength, ksi	59.73	55.75	49.59	
Elongation in 5.08 Cm, %	16.0	17.5	21.0	

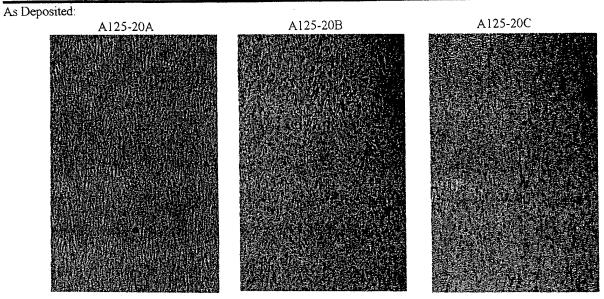


Figure A-8. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample A125-30A	Sample A125-30B	Sample A125-30C	
Nickel Metal, g/l	82.4	82.4	82.4	
Bone Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pH	3.6	3.6	3.6	
Bath Temperature. °C	51.7	51 7	51.7	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.084	0.104	0.117	
Ultimate Strength, ksi	97.89	99.42	79.95	
Yield Strength, ksi	66.87	69.35	50.99	
Elongation in 5.08 Cm, %	8.0	10.0	11.0	
Vickers Hardness, 100 g Load	206	206	193	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.081	0.099	0.114	
Ultimate Strength, ksi	84.47	81.22	73.32	
Yield Strength, ksi	59.01	54.57	42.85	
Elongation in 5.08 Cm, %	8.0	15.5	24.0	

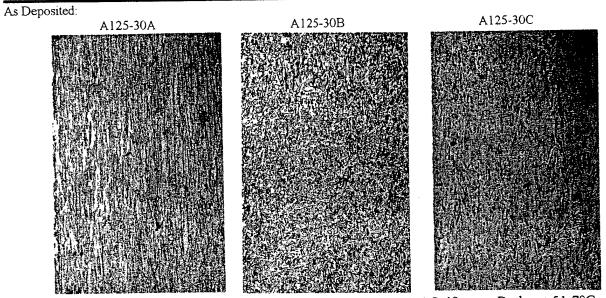


Figure A-9 Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample	B115-20	Sample B115-30	Sample B115-40
Nickel Metal, g/l		82.4	82.4	82.4
Boric Acid, g/l		31.5	31.5	31.5
Chloride, g/l	C	.406	0.406	0.406
Operating Conditions				
pН		4.0	4.0	4.0
Bath Temperature, °C		46.1	46.1	46.1
Filtration, Micron Rating		20	20	20
Current Mode	Conve	entional dc	Conventional dc	Conventional dc
Current Density, A/dm ²		2.15	3.23	4.30
Anodes	S	D Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle		Single Spray Nozzle	Single Spray Nozzle
Physical Properties				
As-Deposited	(1)	(2)		
Thickness, Cm	0.124	0.163	0.102	0.107
Ultimate Strength, ksi	117.80	122.00	87.00	90.48
Yield Strength, ksi	74.85	88.08	61.50	54.29
Elongation in 5.08 Cm, %	12.0	10.0	10.0	8.0
Vickers Hardness, 100 g Load	170	237	247	197
Stress Relieved at 350°C for 1 Hour	ſ			
Thickness, Cm	0.114	0.160	0.099	0.104
Ultimate Strength, ksi	93.32	83.62	77.47	85.67
Yield Strength, ksi	65.90	61.02	54.79	62.92
Elongation in 5.08 Cm, %	20.0	25.0	16.0	10.0

Photomicrographs (100 X Magnification)

As Deposited:
B115-20
B115-30
B115-40

Figure A-10. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 46.1°C Using Conventional Current.

Chemistry	Sample B115-20A	Sample B115-20B	Sample B115-20C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pH	4.0	4.0	4.0
Bath Temperature, °C	46.1	46.1	46.1
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	e Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.112	0.132	0.114
Ultimate Strength, ksi	118.60	123.60	123.10
Yield Strength, ksi	90.09	81.00	76.50
Elongation in 5.08 Cm, %	5.5	11.0	9.0
Vickers Hardness, 100 g Load	213	221	206
Stress Relieved at 350°C for 1 Hou	r		
Thickness, Cm	0.112	0.132	0.119
Ultimate Strength, ksi	93.55	104.40	91.62
Yield Strength, ksi	67.63	67.43	60.66
Elongation in 5.08 Cm, %	19.5	20.0	14.5

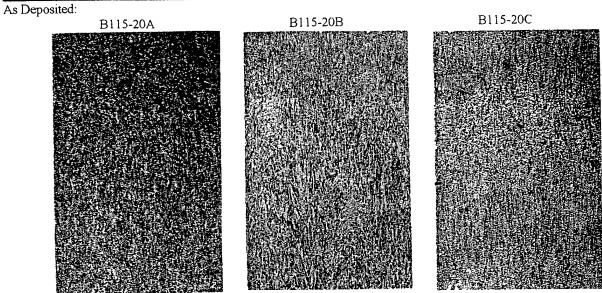


Figure A-11 Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample B115-30A	Sample B115-30B	Sample B115-30C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.()
Bath Temperature, °C	46.1	46.1	46.1
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 30% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	3 On, 7 Off'	8 Forward, 2 Reverse
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.114	0.099	0.124
Ultimate Strength, ksi	74.52	119.50	99.39
Yield Strength, ksi	49.38	77.98	63. 2 9
Elongation in 5.08 Cm, %	15.0	7.5	11.0
Vickers Hardness, 100 g Load	180	193	193
Stress Relieved at 350°C for 1 Hou	r		
Thickness, Cm	0.117	0.102	0.124
Ultimate Strength, ksi	67.08	83.00	86.03
Yield Strength, ksi	45.30	53.68	61.13
Elongation in 5.08 Cm, %	20.0	17.5	11.0

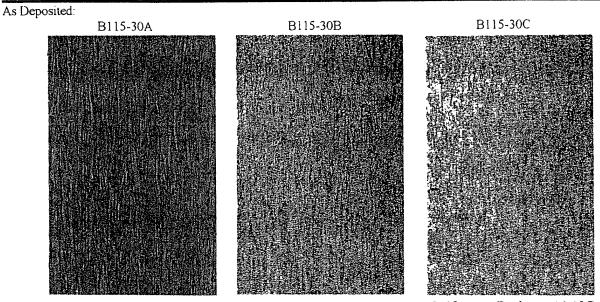


Figure A-12. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample B120-20	Sample B120-30	Sample B120-40	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pН	4.0	4.0	4.0	
Bath Temperature, °C	48.9	48.9	48.9	
Filtration, Micron Rating	20	20	20	
Current Mode	Conventional de	Conventional dc	Conventional dc	
Current Density, A/dm ²	2.15	3.23	4.30	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties		·		
As-Deposited				
Thickness, Cm	0.124	0.119	0.127	
Ultimate Strength, ksi	117.80	128.20	73.31	
Yield Strength, ksi	74.85	90.45	47.81	
Elongation in 5.08 Cm, %	12.0	6.5	16.0	
Vickers Hardness, 100 g Load	170	237	176	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.114	0.119	0.130	
Ultimate Strength, ksi	93.32	92.22	75.88	
Yield Strength, ksi	65.90	67.26	51.75	
Elongation in 5.08 Cm, %	19.5	15.0	17.0	

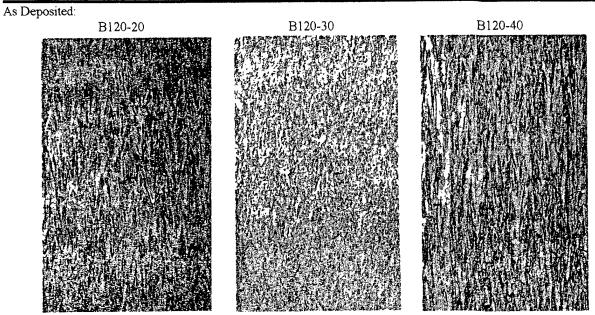


Figure A-13. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 48.9°C Using Conventional Current.

Chemistry	Sample B120-20A	Sample B120-20B	Sample B120-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pH	4.()	4.0	4.0	
Bath Temperature, °C	48.9	48.9	48.9	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle		
Pulse or PR Timing, Msec	5 On, 5 Off	2 On. 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.132	0.132	0.119	
Ultimate Strength, ksi	119.50	111.10	102.80	
Yield Strength, ksi	80.66	80.84	69.37	
Elongation in 5.08 Cm, %	7.5	8.5	14.0	
Vickers Hardness, 100 g Load	237	237	221	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.132	0.130	0.119	
Ultimate Strength, ksi	90.21	77.04	86.90	
Yield Strength, ksi	70.72	50.97	61.89	
Elongation in 5.08 Cm, %	14.0	28.5	14.0	

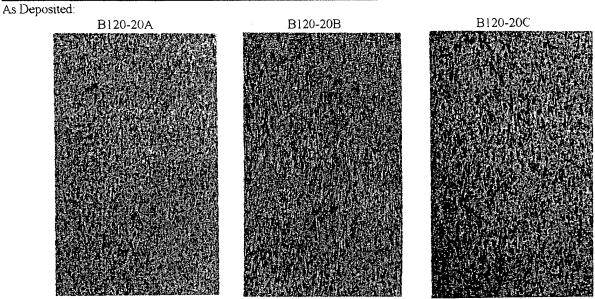


Figure A-14. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample B120-30A	Sample B120-30B	Sample B120-30C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pH	4.0	4.0	4.0
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Mscc	5 On. 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm			
Ultimate Strength, ksi			
Yield Strength, ksi			
Elongation in 5.08 Cm, %			
Vickers Hardness, 100 g Los	ad		
Stress Relieved at 350°C for 1 He	OUL		
Thickness, Cm			
Ultimate Strength, ksi			
Yield Strength, ksi			
Elongation in 5.08 Cm, %			
Photomicrographs (100 X	K Magnification)		
As Deposited:			D100 000
B120-30A	B120	-30B	B120-30C

Figure A-15. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample B125-20	Sample B125-30	Sample B125-40
Nickel Metal, g/l	82.4	82.4	82.4
Bonc Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pH	4.0	4.0	4.0
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional de
Current Density, A/dm²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.048 (Thin)	0.109	0.130
Ultimate Strength, ksi	138.40	97.15	90.84
Yield Strength, ksi	98.42	72.74	71.04
Elongation in 5.08 Cm. %	11.0	7.5	11.5
Vickers Hardness, 100 g Load	237	206	233
Stress Relieved at 350°C for 1 Hou	r	,	
Thickness, Cm	0.051	0.109	0.130
Ultimate Strength, ksi	96.12	86.56	69.10
Yield Strength, ksi	74.20	61.23	41.15
Elongation in 5.08 Cm, %	14.5	19.0	18.0

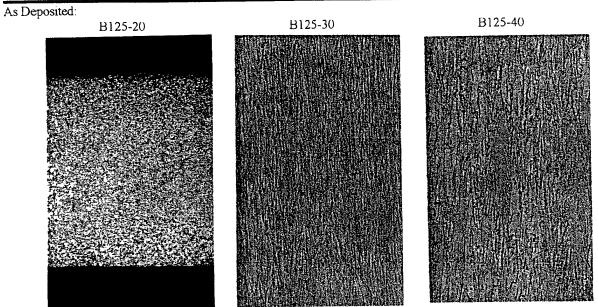


Figure A-16. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 51.7°C Using Conventional Current.

Chemistry	Sample B125-20A	Sample B125-20B	Sample B125-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pН	4.0	4.0	4 ()	
Bath Temperature, °C	51.7	51.7	51 7	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	e Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties	`			
As-Deposited				
Thickness, Cm	0.117	0.117	0.122	
Ultimate Strength, ksi	104.20	110.00	111.20	
Yield Strength, ksi	64.57	75.33	79.46	
Elongation in 5.08 Cm, %	11.5	9.5	10.0	
Vickers Hardness, 100 g Load	221	228	234	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.119	0.122	0.124	
Ultimate Strength, ksi	82.14	84.57	96.97	
Yield Strength, ksi	58.13	61.88	71.92	
Elongation in 5.08 Cm, %	14.0	13.0	16.0	

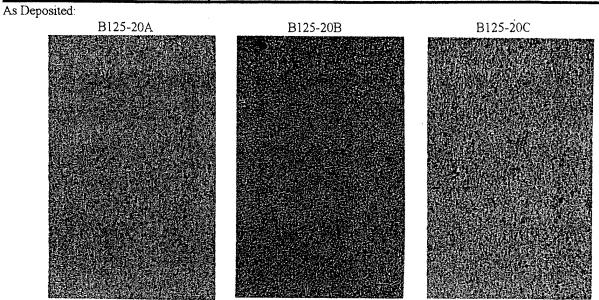


Figure A-17. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 51.7°€ Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample B125-30A	Sample B125-30B	Sample B125-30C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.0
Bath Temperature. °C	51.7	51 7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.107		
Ultimate Strength, ksi	113.40		
Yield Strength, ksi	76.05		
Elongation in 5.08 Cm, %	12.5		
Vickers Hardness, 100 g Load	221		
Stress Relieved at 350°C for 1 Hour	-		
Thickness, Cm	0.109		
Ultimate Strength, ksi	94.14		
Yield Strength, ksi	68.30		
Elongation in 5.08 Cm, %	15.0		

Photomicrographs (100 X Magnification)

As Deposited:

B125-30A

B125-30B B125-30C

Figure A-18. Test Data for Thick Nickel Deposits from pH 4.0 Sulfamate Baths at 51.7°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample C115-20	Sample C115-30	Sample C115-40	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions			·	
pН	4.4	4.4	4.4	
Bath Temperature, °C	46.1	46.1	46.1	
Filtration, Micron Rating	20	20	20	
Current Mode	Conventional dc	Conventional dc	Conventional dc	
Current Density, A/dm ²	2.15	3.23	4.30	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties		•		
As-Deposited				
Thickness, Cm	0.160	0.224	0.130	
Ultimate Strength, ksi	110.60	98.64	85.13	
Yield Strength, ksi	74.28	60.80	59.88	
Elongation in 5.08 Cm, %	10.5	12.5	10.0	
Vickers Hardness, 100 g Load	201	181	181	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.150	0.221	0.124	
Ultimate Strength, ksi	102.50	90.99	91.90	
Yield Strength, ksi	72.54	70.24	71.86	
Elongation in 5.08 Cm, %	17.0	12.5	7.5	

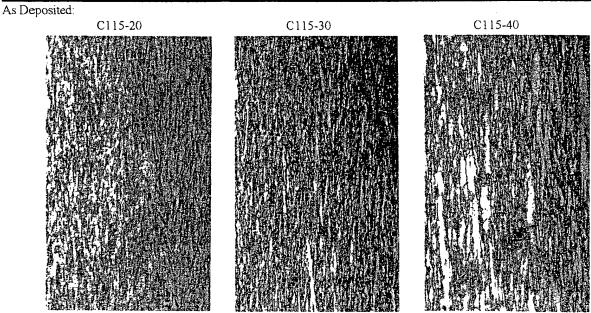


Figure A-19. Test Data for Thick Nickel Deposits from pH 4.4 Sulfamate Baths at 46.1°C Using Conventional Current.

Chemistry	Sample C115-20A	Sample C115-20B	Sample C115-20C	
Nickel Metal, g/l	82.4	82 4	82.4	
Boric Acid. g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions			ړ	
pН	4 4	4.4	4.4	
Bath Temperature, °C	46 1	46.1	46.1	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On. 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average	2 15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Sprav Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.071	0.114		
Ultimate Strength, ksi	119.30	108.90		
Yield Strength, ksi	79.29	65.07		
Elongation in 5.08 Cm. %	9.0	10.0		
Vickers Hardness, 100 g Load	230	228		
Stress Relieved at 350°C for I Hou				
Thickness, Cm	0.071	0 109		
Ultumate Strength, ksi	88.59	92.64		
Yield Strength, ksi	65.56	69.48		
Elongation in 5.08 Cm. %	17.5	11.0		

Photomicrographs (100 X Magnification)

As Deposited:

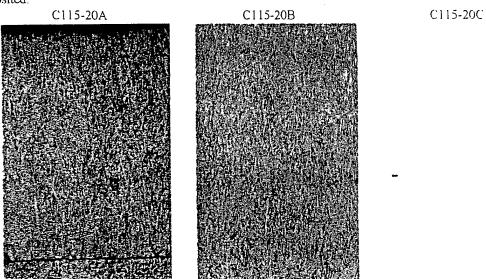


Figure A-20 Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current

Chemistry	Sample C115-30A	Sample C115-30B	Sample C115-30C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pН	3.6	3.6	3.6	
Bath Temperature, °C	. 46.1	46.1	46.1	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 30% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	3 On, 7 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
as-Deposited				
Thickness, Cm	0.097	0.104	0.102	
Ultimate Strength, ksi	123.50	119.80	109.30	
Yield Strength, ksi	79.80	77.60	78.53	
Elongation in 5.08 Cm, %	6.5	8.5	9.5	
Vickers Hardness, 100 g Load	209	221	228	
stress Relieved at 350°C for 1 Hou				
Thickness, Cm	0.099	0.104	0.104	
Ultimate Strength, ksi	87.31	79.58	91.35	
Yield Strength, ksi	66.50	57.54	69.49	
Elongation in 5.08 Cm, %	18.5	23.0	10.5	

Photomicrographs (100 X Magnification)

As Deposited:

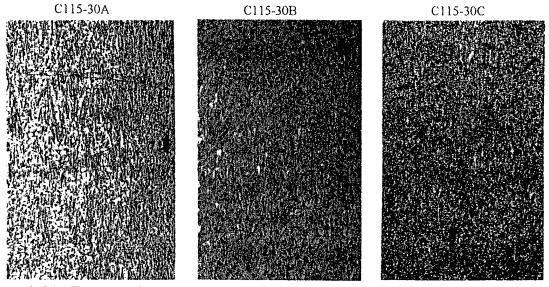


Figure A-21. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Pulsed Current or Periodic Reversed Current.

Chemistry	Sample C120-20	Sample C120-30	Sample	C120-40
Nickel Metal, g/l	82.4	82.4	8	32.4
Boric Acid, g/l	31.5	31.5	3	1.5
Chloride, g/l	0.406	0.406	0.4	406
Operating Conditions				
рН	3.6	3.6		3.6
Bath Temperature, °C	48.9	48.9	4	8.9
Filtration, Micron Rating	20	20		20
Current Mode	Conventional dc	Conventional dc	Conver	ntional de
Current Density, A/dm ²	2.15	3.23	4	.30
Anodes	SD Ni	SD Ni	SD	Ni Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spi	ray Nozzle
Physical Properties				
As-Deposited			(1)	(2)
Thickness, Cm	0.109	0.107	0.114	0.114
Ultimate Strength, ksi	120.70	117.40	93.18	94.97
Yield Strength, ksi	83.22	74.48	66.84	66.70
Elongation in 5.08 Cm, %	9.5	6.5	6.5	7.0
Vickers Hardness, 100 g Load	237	237	190	213
Stress Relieved at 350°C for 1 Hour	r			
Thickness, Cm	0.109	0.107	0.109	0.107
Ultimate Strength, ksi	105.20	98.20	87.22	88.81
Yield Strength, ksi	85.07	73.06	70.14	62.35
Elongation in 5.08 Cm, %	7.5	11.5	6.0	6.5

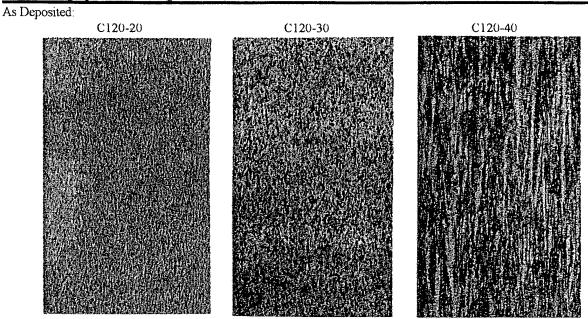


Figure A-22. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

Chemistry	Sample C120-20A	Sample C120-20B	Sample C120-20C	
Nickel Metal, g/l	82.4	82.4	82.4	
Boric Acid, g/l	31.5	31.5	31.5	
Chloride, g/l	0.406	0.406	0.406	
Operating Conditions				
pН	3.6	3.6	3.6	
Bath Temperature, °C	48.9	48.9	48.9	
Filtration, Micron Rating	20	20	20	
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse	
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Reverse	
Anodes	SD Ni	SD Ni	SD Ni	
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle	
Physical Properties				
As-Deposited				
Thickness, Cm	0.114	0.119	0.109	
Ultimate Strength, ksi	90.55	131.19	88.30	
Yield Strength, ksi	62.22	88.22	62.41	
Elongation in 5.08 Cm, %	8.0	8.5	8.5	
Vickers Hardness, 100 g Load	213	274	206	
Stress Relieved at 350°C for 1 Hou	r			
Thickness, Cm	0.122	0.119	0.109	
Ultimate Strength, ksi	80.78	96.23	84.91	
Yield Strength, ksi	57.58	81.39	68.30	
Elongation in 5.08 Cm, %	10.0	10.0	5.0	

Photomicrographs (100 X Magnification)

As Deposited:

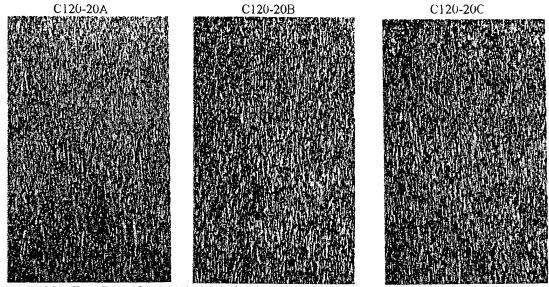


Figure A-23. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THICK NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 4.4

Chemistry	Sample C120-30A	Sample C120-30B	Sample C120-30C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties	Y		
As-Deposited			
Thickness, Cm	0.109		
Ultimate Strength, ksi	113.90		
Yield Strength, ksi	73.65		
Elongation in 5.08 Cm, %	11.0		
Vickers Hardness, 100 g Load	237		
Stress Relieved at 350°C for 1 Hou	ī		
Thickness, Cm	0.109		
Ultimate Strength, ksi	82.06		
Yield Strength, ksi	58.25		
Elongation in 5.08 Cm, %	18.0		

Photomicrographs (100 X Magnification)

As Deposited: C120-30A C120-30B C120-30C

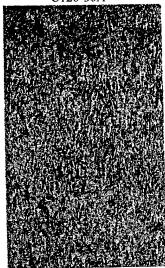


Figure A-24. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Pulsed Current or Periodic Reversed Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THICK NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 4.4

Chemistry	Sample C125-20	Sample C125-30	Sample C125-40
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.109	0.094	0.119
Ultimate Strength, ksi	124.40	114.20	126.30
Yield Strength, ksi	80.08	75.77	86,90
Elongation in 5.08 Cm, %	9.5	10.5	10.0
Vickers Hardness, 100 g Load	221	221	221
Stress Relieved at 350°C for 1 Hour	r		
Thickness, Cm	0.112	0.098	0.124
Ultimate Strength, ksi	84.36	80.02	77.62
Yield Strength, ksi	60.36	54.21	50.51
Elongation in 5.08 Cm, %	21.5	27.0	28.0

Photomicrographs (100 X Magnification)

As Deposited:

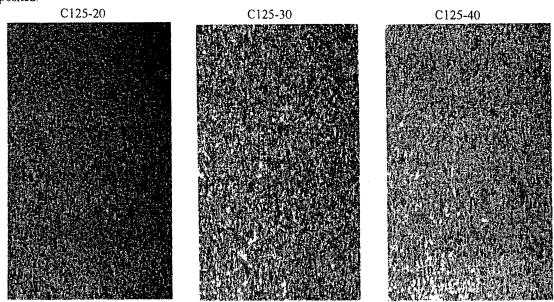


Figure A-25. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Conventional Current.

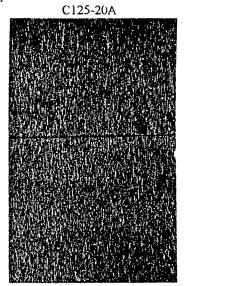
MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THICK NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 4.4

Chemistry	Sample C125-20A	Sample C125-20B	Sample C125-20C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	3.6	3.6	3.6
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Forward, 1.08 Revers
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.099		0.107
Ultimate Strength, ksi	93.59		84.00
Yield Strength, ksi	64.09		56.79
Elongation in 5.08 Cm, %	5.5		8.0
Vickers Hardness, 100 g Load	234		181
Stress Relieved at 350°C for 1 Hour			
Thickness, Cm	0.102		0.109
Ultimate Strength, ksi	84.33		76.75
Yield Strength, ksi	63.49		57.79
Elongation in 5.08 Cm, %	7.5		7.0

C125-20B

Photomicrographs (100 X Magnification)

As Deposited:



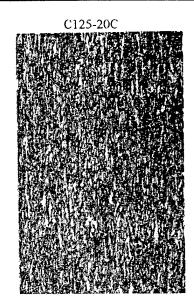


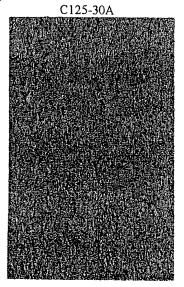
Figure A-26. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Pulsed Current or Periodic Reversed Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THICK NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 4.4

Chemistry	Sample C125-30A	Sample C125-30B	Sample C125-30C
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	3.23 Average	3.23 Average	3.23 Forward, 1.62 Reverse
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited		······································	
Thickness, Cm	0.117		
Ultimate Strength, ksi	100.50		
Yield Strength, ksi	61.80		
Elongation in 5.08 Cm, %	11.5		
Vickers Hardness, 100 g Load	224		
Stress Relieved at 350°C for 1 Hou	r		
Thickness, Cm	0.114		
Ultimate Strength, ksi	87.30		
Yield Strength, ksi	58.64		
Elongation in 5.08 Cm, %	12.5		

Photomicrographs (100 X Magnification)

As Deposited:



C125-30B C125-30C

Figure A-27. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Pulsed Current or Periodic Reversed Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THICK NICKEL DEPOSITS FROM SULFAMATE BATHS AT 48.9°C AND PH OF 3.6, 4.0, 4.4

Chemistry	Sample A120-20D	Sample B120-20D	Sample C120-20D
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.4	4.()	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 50% Duty Cycle	Pulsed, 50% Duty Cycle
Pulse or PR Timing, Msec	0.5 On, 0.5 Off	0.5 On, 0.5 Off	0.5 On, 0.5 Off
Current Density, A/dm ²	2.15 Average	2.15 Average	2.15 Average
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.132	0.127	0.102
Ultimate Strength, ksi	98.05	105.80	90.28
Yield Strength, ksi	71.35	81.91	61.01
Elongation in 5.08 Cm, %	10.0	8.5	9.0
Vickers Hardness, 100 g Load	221	237	221
Stress Relieved at 350°C for 1 Hour	*		
Thickness, Cm	0.130	0.119	0.104
Ultimate Strength, ksi	80.90	87.05	84.87
Yield Strength, ksi	56.92	60.30	66.93
Elongation in 5.08 Cm, %	15.0	17.5	5.5

Photomicrographs (100 X Magnification)

As Deposited:

A120-20D

B120-20D

C120-20D

Figure A-27. Test Data for Thick Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Pulsed Current at a Frequency of 1000 Hz.

APPENDIX B

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR AS-DEPOSITED AND STRESS RELIEVED NICKEL DEPOSITS FROM SULFAMATE BATHS - DEPOSIT THICKNESSES BETWEEN 0.0254 AND 0.0508 CM (0.010 AND 0.020 INCHES)

1998 STUDY IN 136 LITER BATHS

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 46.1°C AND PH OF 3.6

Chemistry	Sample A115-20TW	Sample A115-30TW	Sample A115-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pH	3.6	3.6	3.6
Bath Temperature, °C	46.1	46.1	46.1
Filtration, Micron Rating	20	20	20
Current Mode	Conventional do	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0381	0.0381	0.0368
Ultimate Strength, ksi	84.77	-80.80	83.45
Yield Strength, ksi	58.28	54.97	62.24
Elongation in 5.08 Cm, %	9.5	10.5	7.5
Elongation in 2.54 Cm, %	14.0	15.0	14.0
Stress Relieved at 177°C for 4 F	lours		
Thickness, Cm	0.0356	0.0406	0.0381
Ultimate Strength, ksi	88.86	67.50	80.98
Yield Strength, ksi	64.57	42.22	58.38
Elongation in 5.08 Cm, %	4.5	14.0	5.5
Elongation in 2.54 Cm, %	9.0	21.0	9.0
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0356	0.0406	0.0394
Ultimate Strength, ksi	78.84	70.81	66.67
Yield Strength, ksi	61.08	54.04	48.08
Elongation in 5.08 Cm, %	10.0	10.5	16.0
Elongation in 2.54 Cm, %	16.0	19.0	23.0

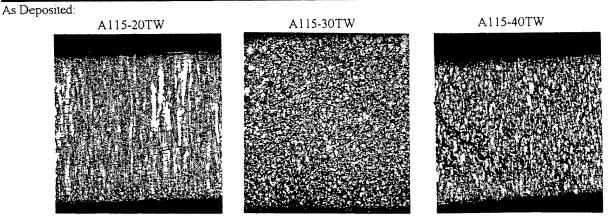


Figure B-1. Test Data for Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 46.1°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 3.6

Chemistry	Sample A120-20TW	Sample A120-30TW	Sample A120-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Conventional de	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0330	0.0381	0.0394
Ultimate Strength, ksi	120.80	99.21	87.18
Yield Strength, ksi	82.57	70.11	59.62
Elongation in 5.08 Cm, %	12.0	6.0	5.0
Elongation in 2.54 Cm, %	17.0	10.0	5.0
Stress Relieved at 177°C for 4 H	lours		
Thickness, Cm	0.0330	0.0356	0.0356
Ultimate Strength, ksi	115.70	113.30	91.16
Yield Strength, ksi	81.54	82.60	68.47
Elongation in 5.08 Cm, %	10.0	4.5	4.0
Elongation in 2.54 Cm, %	15.0	8.0	7.0
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0330	0.0368	0.0368
Ultimate Strength, ksi	91.60	74.56	74.56
Yield Strength, ksi	68.70	45.14	. 54.72
Elongation in 5.08 Cm, %	13.5	22.0	9.0
Elongation in 2.54 Cm, %	23.0	30.0	16.0

Photomicrographs (100 X Magnification)

As Deposited:

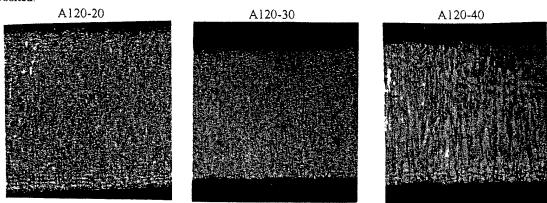


Figure B-2: Test Data for Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 3.6

Chemistry	Sample A125-20TW	Sample A125-30TW	Sample A125-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	3.6	3.6	3.6
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional do
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties		1	
As-Deposited			
Thickness, Cm	0.0406	0.0356	
Ultimate Strength, ksi	106.70	111.20	
Yield Strength, ksi	73.82	83.93	
Elongation in 5.08 Cm, %	5.5	4.5	
Elongation in 2.54 Cm, %	10.0	7.0	
Stress Relieved at 177°C for 4 H	lours		
Thickness, Cm	0.0406	0.0381	
Ultimate Strength, ksi	95.14	95.75	
Yield Strength, ksi	67.25	64.23	
Elongation in 5.08 Cm, %	6.0	6.0	
Elongation in 2.54 Cm, %	9.0	10.0	<u> </u>
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0394	0.0381	
Ultimate Strength, ksi	91.26	81.12	
Yield Strength, ksi	71.34	55.45	
Elongation in 5.08 Cm, %	8.5	12.5	
Elongation in 2.54 Cm, %	12.0	19.0	

Photomicrographs (100 X Magnification)

As Deposited:

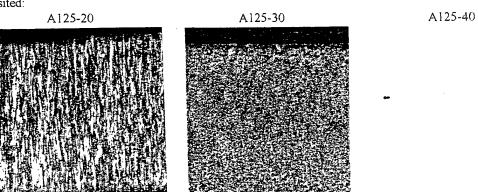
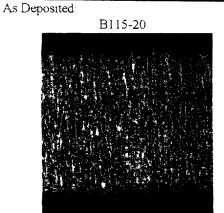


Figure B-3. Test Data for Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 46.1°C AND PH OF 4.0

Chemistry	Sample B115-20TW	Sample B115-30TW	Sample B115-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chlonde, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.0
Bath Temperature, °C	46.1	46.1	46.1
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional de
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0356	0.0356	0.0356
Ultimate Strength, ksi	127.50	101.80	124.00
Yield Strength, ksi	96.32	73.26	82.36
Elongation in 5.08 Cm, %	4.5	2.0	2.0
Elongation in 2.54 Cm, %	9.0	5.0	5.0
Stress Relieved at 177°C for 4 Hou	urs		
Thickness, Cm	0.0356	0.0356	0.0381
Ultimate Strength, ksi	112.70	89.59	94.15
Yield Strength, ksi	79.17	69.90	66.49
Elongation in 5.08 Cm, %	3.5	4.5	5.0
Elongation in 2.54 Cm, %	7.0	6.0	8.0
Stress Relieved at 350°C for 1 Hou	r		
Thickness, Cm	0.0356	0.0330	0.0356
Ultimate Strength, ksi	99.43	95.70	90.01
Yield Strength, ksi	76.71	76.19	64.48
Elongation in 5.08 Cm, %	10.5	2.0	10.0
Elongation in 2.54 Cm, %	18.0	5.0	13.0





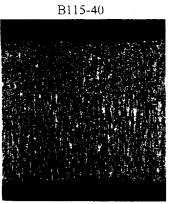


Figure B-4. Test Data for Thin Nickel Deposits from pH 4 0 Sulfamate Baths at 46.1°C Using Conventional Current

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 4.0

Chemistry	Sample B120-20TW	Sample B120-30TW	Sample B120-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.0
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional de
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties	,		
As-Deposited			
Thickness, Cm	0.0356	0.0356	0.0356
Ultimate Strength, ksi	125.71	123.60	101.60
Yield Strength, ksi	93.75	90.20	67.47
Elongation in 5.08 Cm. %	7.5	5.0	7.0
Elongation in 2.54 Cm, %	13.0	10.0	10.0
Stress Relieved at 177°C for 4 H	ours		
Thickness, Cm	0.0381	0.0406	0.0356
Ultimate Strength, ksi	111.20	90.91	90.19
Yield Strength, ksi	76.60	66.00	63.73
Elongation in 5.08 Cm, %	8.5	5.5	6.5
Elongation in 2.54 Cm, %	15.0	8.0	10.0
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0356	0.0381	0.0343
Ultimate Strength, ksi	92.07	83.44	86.77
Yield Strength, ksi	67.99	61.59	63.97
Elongation in 5.08 Cm, %	14.5	13.0	10.0
Elongation in 2.54 Cm, %	25.0	22.0	15.0

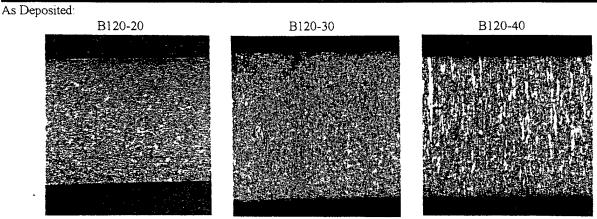


Figure B-5. Test Data for Thin Nickel Deposits from pH 4.0 Sulfamate Baths at 48.9°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 4.0

Chemistry	Sample B125-20TW	Sample B125-30TW	Sample B125-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.0
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional de	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0381	0.0394	0.0374
Ultimate Strength, ksi	125.33	108.11	105.13
Yield Strength, ksi	81.79	73.36	77.56
Elongation in 5.08 Cm, %	10.5	6.0	5.0
Elongation in 2.54 Cm, %	20.0	6.0	10.0
Stress Relieved at 177°C for 4 H	ours		
Thickness, Cm	0.0381	0.0381	0.0406
Ultimate Strength, ksi	122.10	110.70	94.64
Yield Strength, ksi	87.90	78.53	68.58
Elongation in 5.08 Cm, %	12.0	7.0	8.0
Elongation in 2.54 Cm, %	17.0	12.0	6.0
Stress Relieved at 350°C for 1 H	our		
Thickness, Cm	0.0406	0.0432	0.0406
Ultimate Strength, ksi	84.99	74.27	80.65
Yield Strength, ksi	60.79	49.71	59.55
Elongation in 5.08 Cm, %	26.0	15.0	8.0
Elongation in 2.54 Cm, %	30.0	25.0	12.0

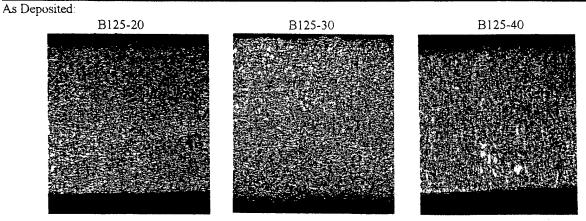


Figure B-6. Test Data for Thin Nickel Deposits from pH 4.0 Sulfamate Baths at 51.7°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 46.1°C AND PH OF 4.4

Chemistry	Sample C115-20TW	Sample C115-30TW	Sample C115-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Bone Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
рН	4.4	4.4	4.4
Bath Temperature, °C	46.1	46.1	46.1
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0356	0.0356	0.0330
Ultimate Strength, ksi	136.10	122.60	116.40
Yield Strength, ksi	94.86	87.77	78.87
Elongation in 5.08 Cm, %	7.0	3.5	4.5
Elongation in 2.54 Cm, %	10.0	6.0	8.0
Stress Relieved at 177°C for 4 H	ours		
Thickness, Cm	0.0330	0.0330	0.0356
Ultimate Strength, ksi	124.70	130.90	103.80
Yield Strength, ksi	86.48	95.39	73.97
Elongation in 5.08 Cm. %	8.0	3.0	4.0
Elongation in 2.54 Cm, %	11.0	5.0	6.0
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0330	0.0381	0.0381
Ultimate Strength, ksi	99.54	90.27	80.32
Yield Strength, ksi	74.50	70.40	61.97
Elongation in 5.08 Cm, %	12.5	7.5	4.5
Elongation in 2.54 Cm, %	20.0	13.0	7.0

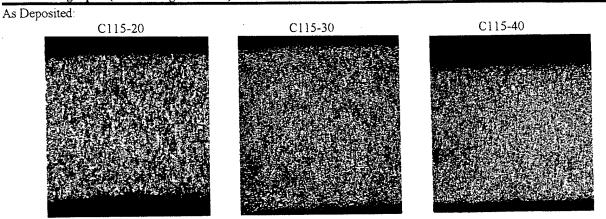
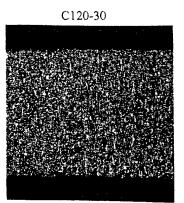


Figure B-7. Test Data for Thin Nickel Deposits from pH 4.4 Sulfamate Baths at 46.1°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 3.6

Chemistry	Sample C120-20TW	Sample C120-30TW	Sample C120-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid. g/l	31 5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0381	0.0343	0.0305
Ultimate Strength, ksi	106.00	124.40	126.91
Yield Strength, ksi	75.50	81.00	85.63
Elongation in 5.08 Cm, %	6.5	8.0	4.5
Elongation in 2.54 Cm, %	12.0	12.0	9.0
Stress Relieved at 177°C for 4 H	ours		
Thickness, Cm	0.0356	0.0356	0.0356
Ultimate Strength, ksi	102.70	109.00	114.30
Yield Strength, ksi	74.47	84.17	82.86
Elongation in 5.08 Cm, %	4.5	2.5	3.0
Elongation in 2.54 Cm, %	8.0	4.0	5.0
Stress Relieved at 350°C for 1 H	lour		
Thickness, Cm	0.0381	0.0356	0.0330
Ultimate Strength, ksi	93.92	92.33	91.60
Yield Strength, ksi	71.43	70.31	67.18
Elongation in 5.08 Cm, %	15.0	17.5	15.0
Elongation in 2.54 Cm, %	22.0	24.0	20.0





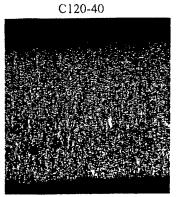


Figure B-8. Test Data for Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 3.6

Chemistry	Sample C125-20TW	Sample C125-30TW	Sample C125-40TW
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	3.6	3.6	3.6
Bath Temperature, °C	51.7	51.7	51.7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional de	Conventional dc	Conventional dc
Current Density. A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
s-Deposited			
Thickness, Cm	0.0279	0.0318	0.0343
Ultimate Strength, ksi	125.68	124.20	129.25
Yield Strength, ksi	84.99	82.01	84.93
Elongation in 5.08 Cm. %	10.0	10.0	9.0
Elongation in 2.54 Cm, %	15.0	17.0	13.0
tress Relieved at 177°C for 4 H	ours		
Thickness, Cm	0.0330	0.0330	0.0356
Ultimate Strength, ksi	113.70	112.10	110.40
Yield Strength, ksi	78.15	77.11	78.75
Elongation in 5.08 Cm, %	9.5	9.5	5.0
Elongation in 2.54 Cm, %	15.0	16.0	8.0
tress Relieved at 350°C for 1 H	our		
Thickness, Cm	0.0279	0.0318	0.0330
Ultimate Strength, ksi	98.78	98.57	91.74
Yield Strength, ksi	73.10	75.52	71.10
Elongation in 5.08 Cm, %	13.5	12.0	13.0
Elongation in 2.54 Cm, %	21.0	16.0	24.0

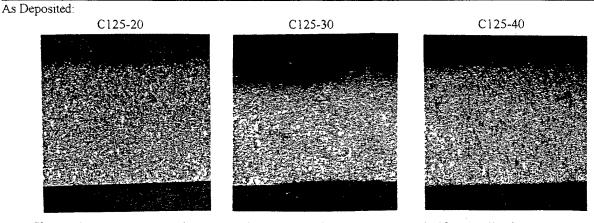


Figure B-9. Test Data for Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 51.7°C Using Conventional Current

APPENDIX C

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR AS-DEPOSITED AND STRESS RELIEVED NICKEL DEPOSITS FROM SULFAMATE BATHS - DEPOSIT THICKNESSES BETWEEN 0.0102 AND 0.0152 CM (0.004 AND 0.006 INCHES)

1998 STUDY IN 136 LITER BATH

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 3.6

Chemistry	Sample A120-20WG	Sample A120-30WG	Sample A120-40WG
	82.4	82.4	82.4
Nickel Metal. g/l	31.5	31.5	31.5
Borne Acid. g/l	0.406	0.406	0.406
Chlonde, g/l Operating Conditions	0.400		
	3.6	3.6	3.6
pН	3.0 48.9	48.9	48.9
Bath Temperature. °C	· · ·	20	20
Filtration, Micron Rating	20	Conventional dc	Conventional dc
Current Mode	Conventional de	3.23	4.30
Current Density, A/dm ²	2.15	3.23 SD Ni	SD Ni
Anodes	SD Ni		Single Spray Nozzle
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Shighe Spray Nozzie
Physical Properties			
As-Deposited		0.0143	
Thickness, Cm	0.0135	0.0142	
Ultimate Strength, ksi	106.80	100.40	
Yield Strength, ksi	74.53	74.46	
Elongation in 5.08 Cm, %	8.0	5 ()	
Elongation in 2.54 Cm, %			
Stress Relieved at 350°C for 1	Hour		
Thickness. Cm	0.0132	0 0142	
Ultimate Strength. ksi	80.62	75.90	
Yield Strength, ksi	52.71	55.76	
Elongation in 5.08 Cm, %	22.5	11.5	
Elongation in 2.54 Cm, %			·
Photomicrographs (200			
As Deposited:			A120-20CWG
A120-20AW	G 4120	A120-20BWG	
-X120-20-XVV	0=	_ :	

Figure 1 Test Data for Very Thin Nickel Deposits from pH 3 6 Sulfamate Baths at 48.9°C Using Conventional Current

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 3.6

Chemistry	Sample A120-20AWG	Sample A120-20BWG	Sample A120-20CWG
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pH	. 3.6	3.6	3.6
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Pulsed, 50% Duty Cycle	Pulsed, 20% Duty Cycle	Periodic Reversed
Pulse or PR Timing, Msec	5 On, 5 Off	2 On, 8 Off	8 Forward, 2 Reverse
Current Density, A/dm ²	2.15 Average	2.15	2.15
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm			0.0140
Ultimate Strength, ksi			91.54
Yield Strength, ksi			59.19
Elongation in 5 08 Cm, %			9.0
Elongation in 2.54 Cm, %			
Stress Relieved at 350°C for 1	Hour		
Thickness, Cm			0.0145
Ultimate Strength, ksi			77.39
Yield Strength, ksi			55.83
Elongation in 5.08 Cm, %			9.0
Elongation in 2.54 Cm, %			
Photomicrographs (200	X Magnification)		
As Deposited:			
A120-20AW	G A120-20BWG		A120-20CWG

Figure 2. Test Data for Very Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 51.7°C AND PH OF 3.6

Chemistry	Sample A125-20WG	Sample A125-30WG	Sample A125-40WG
Nickel Metal. g/l	82.4	82.4	82.4
Boric Acid. 2/1	31.5	31.5	31.5
Chloride, g/l	0.406	0.406	0.406
Operating Conditions			
pН	3.6	3.6	3.6
Bath Temperature. °C	51 7	51.7	5! 7
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional dc	Conventional dc
Current Density, A/dm ²	2.15	3.23	4 30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0122		
Ultimate Strength, ksi	109.20		
Yield Strength, ksi	79.50		
Elongation in 5.08 Cm.	% 9.5		
Elongation in 2.54 Cm,	%		
Stress Relieved at 350°C for	1 Hour		
Thickness. Cm	0.0122		
Ultimate Strength, ksi	77.82		
Yield Strength, ksi	54.81		
Elongation in 5.08 Cm.	% 21.5		
Elongation in 2.54 Cm,	%		
Photomicrographs (20	00 X Magnification)		
As Deposited:			
A125-20W	'G A125-	30WG	A125-40WG

Figure 3 Test Data for Very Thin Nickel Deposits from pH 3.6 Sulfamate Baths at 48.9°C Using Conventional Current.

MECHANICAL PROPERTIES AND DEPOSITION PARAMETERS FOR THIN NICKEL DEPOSITS FROM SULFAMATE SOLUTIONS AT 48.9°C AND PH OF 4.0

Chemistry	Sample B120-20WG	Sample B120-30WG	Sample B120-40WC
Nickel Metal, g/l	82.4	82.4	82.4
Boric Acid, g/l	31 5	31.5	31.5
Chloride, g/I	0.406	0.406	0.406
Operating Conditions			
pН	4.0	4.0	4.0
Bath Temperature, °C	48.9	48.9	48.9
Filtration, Micron Rating	20	20	20
Current Mode	Conventional dc	Conventional de	Conventional do
Current Density, A/dm ²	2.15	3.23	4.30
Anodes	SD Ni	SD Ni	SD Ni
Solution Agitation	Single Spray Nozzle	Single Spray Nozzle	Single Spray Nozzle
Physical Properties			
As-Deposited			
Thickness, Cm	0.0127	0.0127	
Ultimate Strength, ksi	124.50	124.10	
Yield Strength, ksi	90.76	87.15	
Elongation in 5.08 Cm, 9	7.5	6.5	
Elongation in 2.54 Cm, 9			
Stress Relieved at 350°C for	l Hour		
Thickness, Cm	0.0130	0.0127	
Ultimate Strength, ksi	86.96	97.19	
Yield Strength, ksi	63.64	78.31	
Elongation in 5.08 Cm,		12.5	
Elongation in 2.54 Cm, 9	<u>/o</u>		
Photomicrographs (20	0 X Magnification)		
As Deposited:			
B120-20	WG B1	B120-30WG	

Figure 4. Test Data for Very Thin Nickel Deposits from pH 3 6 Sulfamate Baths at 48.9°C Using Conventional Current.

REPORT DOCUMENTATION PAGE

FOITH Approved
OM8 No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden. To Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Artington, VA 222024302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, OC 20503.

Davis Highway, Suite 1204, Arlington, VA 22202-4302		dget, Paperwork Reduction Projec	t (0704-0188), Washington, DC 20503,
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND	•
	14 July 1999 Final June 1998 to		
Innovative Fabrication Techniques for Aerospace			NAS3-98104
G.AUTHOR(S) G.A. Malone Wanda Hudson Rich Edwards Brian Babcock			
7. PERFORMING ORGANIZATION NAME	(S) AND ADDRESS(ES)		B. PERFORMING ORGANIZATION
Electroformed Nickel, In 785 Martin Road Huntsville, AL 35824			REPORT NUMBER
Clevela	e Organization tion Research Progra	Ì	10. SPONSORING / MONITORING AGENCY REPORT NUMBER
11. SUPPLEMENTARY NOTES Work funded by Small Bus Manager, Robert Jankovsk		search Contract 1	NAS3-98104. Project
12a. DISTRIBUTION/AVAILABILITY STATURE Unclassified - Unlimited			12b. DISTRIBUTION CODE
13. ABSTRACT (Maximum 200 words) To be economically attractive, weight and performance for small earth—to—orbit launch systems and station keeping space vehicles must be improved at significantly lower costs while maintaining required payloads. A major weight and cost factor in any of these devices is the tankage for propellants and pressurant gases Innovative and proven manufacturing technologies such as electroforming have been overlooked as means to meet these needs. Electroforming offers the means to fabricate seamless tanks with no property degrading welds. In conjunction with filament winding electroforming can produce improved tanks in greatly reduced time frames, because it can produce the material and the net shape simultaneously. In this study, it has been shown that pure nickel electrodeposits in very small thicknesses suitable for tank line can be made with ample ductility for expansion as a sealant against outer shells of hic modulus such as filament wound graphite fibers. Ability to electroform seamless thin—wall tanks with integrated fill/drain ports has been shown. Electroform joining of tar components without thermal means has been proven. Titanium deposition of coherent film needs further work, but feasible electrolytes have been established. Bonding of nickel to carbon composits was demonstrated.			
14. SUBJECT TERMS Electroformed Tank Liners Nickel Deposit Properties Fabricating Propellant Ta	Composite I	lectrodeposition Pressure Vessels and Structures	
OF REPORT	SECURITY CLASSIFICATION OF THIS PAGE classified	19. SECURITY CLASSIFIC OF ABSTRACT Unclassified	CATION 20. LIMITATION OF ABSTRACT UL